10.2478/v10048-008-0030-y
MEASUREMENT SCIENCE REVIEW, Volume 8, Section 3, No.5, 2008

Thermal Synthesis of Bismuth-Doped Yttrium Iron Garnet for Magneto-Optical Imaging

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This contribution deals with the preparation and characterization of bismuth-doped yttrium iron garnets with composition $BiY_2Fe_5O_{12}$ (Bi:YIG). The samples have been prepared by sintering of metal oxides homogenized in acetone for 10 minutes. The estimated average crystallite size of the single phase Bi:YIG synthesized at temperature 950 °C for 15 hours is ~ 65 nm. The real content of Bi^{3+} cations in samples was quantitatively evaluated by application of real-coded genetic algorithm to the powder X-ray diffraction patterns.

Keywords: bismuth-doped yttrium iron garnet, synthesis, TGA/DTA, XRD, real-coded genetic algorithm

1. INTRODUCTION

THE MAGNETO-OPTICAL METHOD based on Faraday effect is widely used for imaging vortices in high-temperature superconductors [1]. The main component, that affects the resolution of this method, is magneto-optical indicator. It is based on Bi: YIG thin film on gadolinium gallium garnet (GGG) with reflection (Al) and protection (Ti_3N_4) layer [2]. The bismuth substituted ferrimagnetic rare earth iron garnets ($Bi_xR_{3-x}Fe_5O_{12}$, R = Y, Lu, Gd) are the materials with strongly enhanced Faraday rotation [3]. Due to the fact that an increasing of bismuth doping level results in the increase of the Verdet constant value, the aim of this work was to prepare the Bi-doped yttrium iron garnet with relatively high content of bismuth atoms ($x = 1$).

In this paper, we present the technology for preparation of bismuth-doped yttrium iron garnets (Bi:YIG) with nominal composition of $BiY_2Fe_5O_{12}$. The samples were characterized by X-ray powder diffraction (XRD) analysis. The real content of Bi^{3+} cations in samples was quantitatively evaluated by application of genetic algorithm by using nonbinary string encoding to the powder X-ray diffraction patterns.

2. SUBJECT AND METHODS

There are several techniques of preparation of yttrium iron garnets (YIG) and bismuth-doped yttrium iron garnets (Bi:YIG); main techniques are: sintering of iron and yttrium oxides in appropriate molecular ratios [4], using precipitation reaction of iron, yttrium and bismuth chlorides or nitrates [5]–[6], and using sol-gel methods [7]–[11].

The fundamentals of the sintering synthesis method is the chemical reaction

$$(3-x)\ Y_2O_3 + 5\ Fe_2O_3 + x\ Bi_2O_3 \xrightarrow{T,\ t} 2\ Bi_xY_{3-x}Fe_5O_{12} \qquad (1)$$

where

x is the nominal content of bismuth

t is the sintering time ($t = 2$ hours for $x = 0.0$ [4]),

T is the sintering temperature ($T = 1200$ °C for $x = 0.0$ [4]).

The appropriate molecular ratios of iron, yttrium and bismuth oxides are homogenized, pressed into tablets and sintered at temperature T for time t.

For magneto-optical imaging it is very important to obtain the Bi-doped yttrium iron garnet with high content of bismuth, because the increase of bismuth doping level results in the increase of the Verdet constant value. However, the melting point of bismuth oxide is only 817 °C; so the actual content of bismuth in the samples prepared at temperature of 1200 °C can be slightly different from the nominal one. Due to this fact, the sintering temperature T and time t need to be optimized for the nominal content of $x = 1.0$.

The qualitative phase pre-identification of powders was performed by XRD and compared with XRD data of the $BiY_2Fe_5O_{12}$ model created from XRD data of $Bi_{2.88}Fe_5O_{12}$ and $Y_3Fe_5O_{12}$ included in Inorganic Crystal Structure Database (ICSD), by linear assumptions so that the value of the relative intensity or the value of the angle 2θ for each peak for all values x of Bi content is considered to be lying on the straight line crossing two points determined by the values of the relative intensities or the values of the angle 2θ of $Bi_{2.88}Fe_5O_{12}$ and $Y_3Fe_5O_{12}$, respectively.

To evaluate quantitatively the real content of Bi^{3+} cations, we assumed that the value of 2θ is fulfilling the following equation

$$2\theta_j = 2.\arcsin\frac{a_j}{b_j.x + c_j}, \quad \text{for} \quad 0 \le x \le 2.88, \qquad (2)$$

where a_j, b_j, c_j are constants for j-th XRD diffraction peak. Eq. (2) is based on the fact that the lattice constant of $Bi_xY_{3-x}Fe_5O_{12}$ depends linearly on the content of bismuth atoms x [12]. We used Genetic Algorithm [13] with real-coded strings for genomes with the following evaluation function

$$E:(x) \rightarrow \sum_{j=1}^{n} (2\theta_j^{meas} - 2\theta_j)^2, \qquad (3)$$

where

n is the number of used peaks ($n = 18$),

$2\theta_j^{meas}$ is the j-th diffraction angle obtained from XRD diffraction pattern,

$2\theta_j$ is the j-th diffraction angle computed from (2).

The parameters of Genetic Algorithm were set as follows:

number of generations = 100, total number of genomes = 10,000, number of genomes allowed to reproduce = 5,000, the mutation probability = 0.3 and the mate probability = 0.5. The constant values of a_j, b_j, c_j for each peak computed from XRD data of $Bi_{2.88}Fe_5O_{12}$ and $Y_3Fe_5O_{12}$ from ICSD are shown in Tab.1.

Tab.1 The constant values of a_j, b_j, c_j for each peak computed from XRD data of $Bi_{2.88}Fe_5O_{12}$ and $Y_3Fe_5O_{12}$

j	$2\theta j$ [deg] $Bi_{2.88}Fe_5O_{12}$	$2\theta j$ [deg] $Y_3Fe_5O_{12}$	a_j	b_j	c_j
1	31.62	32.32	0.218384492	0.005872445	0.784650763
2	34.72	35.5	0.26197558	0.006489765	0.859318658
3	28.2	28.84	0.174720822	0.00541296	0.701611234
4	26.36	26.94	0.152962615	0.004925187	0.656671718
5	19.86	20.28	0.087435677	0.003609117	0.496641251
6	17.18	17.54	0.065586527	0.00310561	0.430164779
7	38.98	39.86	0.327540311	0.007229505	0.960889917
8	44.1	45.12	0.414801934	0.008235136	1.081196845
9	54.24	55.52	0.611491406	0.00991335	1.312864192
10	52.1	53.34	0.567692298	0.009695843	1.264767338
11	49.92	51.08	0.523973583	0.009155679	1.215318057
12	48.8	49.94	0.502241521	0.009039232	1.189740758
13	53.18	54.44	0.589643614	0.009805342	1.289096702
14	58.32	59.72	0.698668609	0.010632284	1.403280362
15	57.32	58.7	0.67702499	0.010532272	1.381279729
16	67.86	69.56	0.916993937	0.012247272	1.607557323
17	66.02	67.66	0.873500333	0.011945239	1.568981959
18	69.68	71.44	0.960570014	0.012537948	1.645305693

The average crystallite size of the powders was determined using the X-ray broadening of the (420) diffraction peak by Scherrer equation [11]

$$D = \frac{0.9\lambda}{\beta.\cos\theta}, \tag{4}$$

where

D is crystallite size in nm,

λ is the radiation wave length (0.15405 nm for Cu-K_α),

β is the full width at half maximum corrected for instrumental broadening,

θ is the diffraction angle.

3. Results and discussion

The samples with nominal composition $BiY_2Fe_5O_{12}$ were synthesized by conventional solid state reaction of stoichiometric mixtures of Bi_2O_3, Fe_2O_3 and Y_2O_3 powders as described in (1). The samples were homogenized in agate boat for 10 minutes in acetone and then pressed into pellets with diameter 12 mm and 30 mm under the pressure of 200 MPa and 128 MPa, respectively. The pellets were sintered at 950 °C for 15 hours in static air. Fig.1 shows the schematic illustration of the heating schedule.

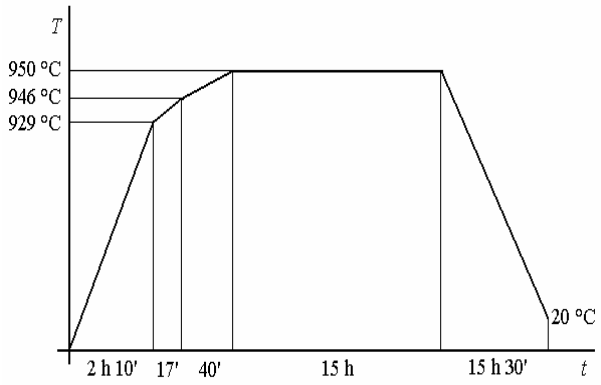

Fig.1 Schematic illustration of heating schedule for $BiY_2Fe_5O_{12}$ sintered at 950 °C.

Fig.2 shows the XRD patterns of the prepared samples and of the model created from XRD data of $Bi_{2.88}Fe_5O_{12}$ and $Y_3Fe_5O_{12}$. The XRD patterns of samples indicate that the powders are a single phase Bi:YIG with the real content of bismuth cations of 0.968. However, in this method we did not take into account the influence of the absorption coefficient. The average crystallite sizes of the synthesized powders found

out from XRD patterns via Scherrer equation in (4) is ~ 65 nm.

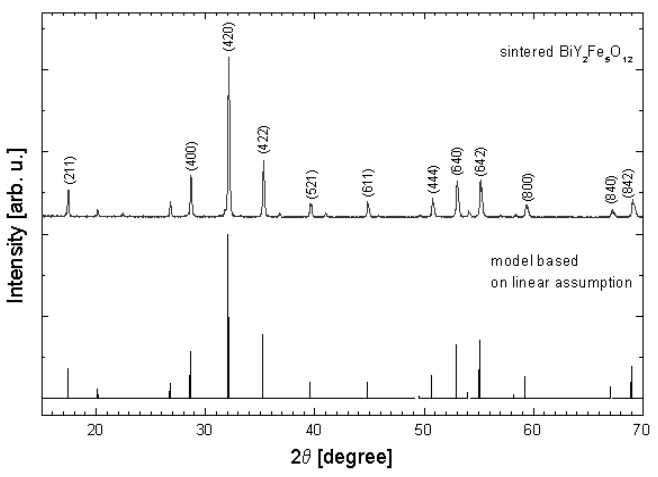

Fig.2 XRD patterns of sintered samples $BiY_2Fe_5O_{12}$ and of a model based on linear assumptions.

4. CONCLUSIONS

Single-phase bismuth-doped yttrium iron garnets were synthesized at 950 °C for 15 hours by sintering of metal oxides. The determined average crystallite size of ~ 65 nm is in accordance with the work of Vajargah et al. [11]. The real content of Bi^{3+} cations in samples was quantitatively evaluated by application of real-coded genetic algorithm to the powder X-ray diffraction patterns to be approximately $x = 0.968$.

ACKNOWLEDGMENT

This work was supported by the Slovak Grant Agency for Science contract number VEGA 2/7083/27 and VEGA 2/7084/27 and the Slovak Research and Development Support Agency, Contract No. APVV-51-059005.

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